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Development of an Unconfined Freeze-Thaw Test for Coarse Aggregates

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DEVELOPMENT OF AN UNCONFINED FREEZE-THAW TEST FOR COARSE AGGREGATE

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Abstract:

Aggregates for use in asphaltic and Portland cement concrete are currently tested in the sulphate soundness test. This test is intended to simulate the action of freezing and thawing on aggregate particles. The precision of this test is poor, and it does not have an excellent correlation with field performance. As a result, The Ministry of Transportation has been developing an unconfined freeze-thaw test for coarse aggregate. This paper describes the test procedure and the ruggedness testing that was done during the development. Both the concentration of the solution and the temperature of freezing have a critical effect on the efficiency of the test. Lower and higher salt concentrations result in reduced loss in the test. Lower temperatures of freezing than -18 C also reduce the amount of aggregate loss in the test. Results of a multilaboratory study of this test are also described. The within-laboratory and multilaboratory precision of this test is significantly better than that of the magnesium sulphate soundness test. Data on the correlation with field performance is also presented that show a better correlation than that obtained with the sulphate soundness test. Test procedures are appended.

Résumé:

À l'heure actuelle, les agrégats utilisés dans la fabrication de ciment asphaltique et de ciment portland sont évalués au moyen d'un essai d'expansion au sulfate. Cet essai vise à simuler les effets du gel et du dégel sur les particules d'agrégats. L'essai n'est pas guère précis et ne correspond pas très bien aux résultats mesurés sur le terrain. Par conséquent, le Ministère des Transports a conçu un test non limité de gel-dégel pour les gros agrégats. Le présent document décrit le déroulement de ce test et le processus d'évaluation de la solidité utilisé lors de la conception du test. La concentration de la solution et la température de congélation ont un effet critique sur l'efficacité du test. Une baisse et une hausse des concentrations en sel entraînent une réduction des pertes lors du test. De plus, on a également remarqué une réduction des pertes d'agrégats lorsque la température de congélation était inférieure à -18 C. Le présent document comprend aussi les résultats d'une étude de ce test effectuée par plusieurs laboratoires. La précision obtenue en laboratoire et dans d'autres laboratoires est beaucoup plus grande que celle de l'essai d'expansion au sulfate de magnésium. On trouvera également les données de corrélation avec les résultats sur le terrain selon lesquelles cette corrélation est supérieure à celle obtenue avec l'essai d'expansion au sulfate. La description du déroulement du test figure en annexe.

Keywords: Freeze-thaw, coarse aggregate, magnesium sulphate soundness, field performance, concrete, asphalt, field performance relationships.

Notes: Test procedure is calibrated using standard aggregate of known freeze-thaw loss.

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ABSTRACT

Aggregates for use in asphaltic and Portland cement concrete are currently tested in the sulphate soundness test. This test is intended to simulate the action of freezing and thawing on aggregate particles. The precision of this test is poor, and it does not have an excellent correlation with field performance. As a result, the Ministry of Transportation has been developing an unconfined freeze-thaw test for coarse aggregate. This paper describes the test procedure and the ruggedness testing that was done during the development. Both the concentration of the solution and the temperature of freezing have a critical effect on the efficiency of the test. Lower and higher salt concentrations result in reduced loss in the test. Lower temperatures of freezing than -18°C also reduce the amount of aggregate loss in the test. Results of multilaboratory study of this test are also described. The within-laboratory and multilaboratory precision of this test is significantly better than that of the magnesium sulphate soundness test. Data on the correlation with field performance is also presented that show a better correlation than that obtained with the sulphate soundness test. The test procedures are appended.

DEVELOPMENT OF AN UNCONFINED FREEZE-THAW TEST FOR COARSE AGGREGATES

INTRODUCTION

In many areas of Canada, especially near urban areas, it is recognized that construction aggregates are a depleting resource. The sterilization of resources by restrictive zoning regulations, building development, and NIMBY (not in my back yard) groups further contribute to shortages of moderate cost aggregates. As a result, there is pressure to use lower quality aggregate sources. At the same time, there is a demand from engineers to use higher quality aggregates than were used in the past. For instance, where higher truck tire pressures and axle loads are causing rutting of asphalt pavements made with natural sand and gravel aggregates, the solution is to use only quarried aggregates in these pavements. Higher traffic volumes call for the use of pavement surfacing aggregates of better frictional properties. High alkali levels in Eastern Canadian Portland cements call for the use of non alkali-reactive aggregates which are not always locally available. There is, thus, a conflict between development, which tends to reduce aggregate resources, and the increasing demand for high quality aggregates as a result of this development.

Aggregate testing and evaluation procedures and specifications have not kept pace with modern technology. The testing procedures currently used are inappropriate for the correct evaluation of the suitability of aggregates. Aggregates are rejected for certain uses when they fail to meet one or more arbitrary specifications, yet the actual field performance may be satisfactory. The automatic rejection of aggregates which fail to meet existing specification requirements leads to a reduction in available resources.

One of the most commonly used aggregate tests in Canada is the magnesium sulphate soundness test. The forerunner of the present magnesium sulphate soundness test was developed in 1828 by M. Brard. The purpose was to simulate the forces generated by freezing water in building stone. In the early nineteenth century, there was no means of freezing water in the laboratory so this test served well. In a recent survey of North American highway authorities, it was found that thirty-five used sulphate soundness tests. Only seven were using freezing and thawing tests (Vogler and Grove, 1987). Aggregates are rarely exposed to the conditions simulated by the sulphate soundness test (crystallization of soluble sulphate salts during oven drying), yet are commonly exposed to freezing and thawing. The continued use of the sulphate soundness test is even more surprising, given the notoriously poor correlation with field performance of aggregates. The only systematic work on the correlation between the magnesium sulphate soundness test and field performance in Canada was that done by Bayne (1949). The performance of aggregate in concrete was evaluated by studying many concrete pavements in Southwestern Ontario. The aggregates were classified into groups: satisfactory, questionable, and unsatisfactory, based on the degree of deterioration of the pavements in which they were incorporated. Bayne found that the range of magnesium sulphate soundness test loss shown by the satisfactory and unsatisfactory groups overlapped, but the mean loss shown by the unsatisfactory aggregates was three times that shown by the satisfactory aggregates. Bayne considered that the limited correlation which they observed was insufficient to be able to apply rigid specification limits without including unsatisfactory aggregate or excluding satisfactory aggregate from use.

Other workers have found similar lack of good correlation. Goldbeck (discussion in Cantrill and Campbell, 1939) noted that the results Cantrill and Campbell presented were another case of the lack of correlation between the sulphate soundness test and field performance. Sweet (1948) found that the sodium sulphate soundness test he performed did not reflect the performance of coarse aggregate in concrete. Vollick and Skillman (1952) found that aggregate used in concrete cylinders which had a high freeze-thaw loss also had a high sodium sulphate soundness test loss. The correlation they obtained showed a wide scatter, indicating that other factors were important in concrete deterioration in addition to deterioration of aggregate in the soundness test.

Larson et al (1964) believed that sulphate soundness testing was not a reliable procedure for predicting the durability of aggregate when confined in concrete. They concluded that no further effort should be directed toward improving this test, and that its use should be discontinued as soon as a suitable alternative could be found. Bloem (1966), in a review of the sulphate soundness test, considered that there was little support for the assumption that performance in the test simulated exposure to freezing and thawing in concrete or that it provided a reliable indication of field performance. He concluded that a low loss was usually, but not always, evidence of good durability, whereas high loss placed an aggregate in a questionable category until service performance was known.

Sturrup (1970) noted several instances in which the sulphate soundness test failed to correlate with field performance in Ontario Hydro structures. He concluded that the application of rigid specification limits for accepting or rejecting aggregate was not justified.

In a study of the field performance of asphalt pavements, Gandhi and Lytton (1984) found a poor correlation between field performance and the sodium sulphate soundness losses of coarse aggregate. They also found no relationship between suitability of asphalt surface course aggregates and their performance in the Los Angeles abrasion and impact test. They concluded that aggregates should not be rejected based on results from these tests alone, and recommended the use of more appropriate tests such as petrographic examination and investigations of service record.

The limited correlation of the sulphate soundness test with field performance is recognized by the standards body responsible for the procedure. ASTM (C88-83) in the introduction to the test states that it "... furnishes information helpful in judging the soundness of aggregates when adequate information is not available from service records of the material exposed to actual weathering conditions". Clearly, the intention is that results should not be compared with rigid specifications, but an element of judgement exercised. Unfortunately, this is rarely the case. In Ontario at least, there are many cases of rejection of aggregates of high loss in this test when the actual field performance is excellent. Sandy dolostones used successfully for asphalt paving courses provide superior frictional properties, and have excellent frost resistance, yet typically give soundness losses in the range of 12 to 17 percent, well outside the normal specification of 5 percent for this quality of aggregate. The unquestioned rejection of aggregates based on their performance in the sulphate soundness test contributes to shortages of aggregates for particular uses. In the case of the sandy dolostone, recognition that field performance was excellent, despite high loss in the test had a major impact on increasing supplies of high quality asphalt paving aggregates in Eastern Ontario in the 1980's.

The sulphate soundness test also suffers from a very poor multilaboratory precision, or reproducibility. A common specification limit is a maximum loss in this test of 12 percent. ASTM reports a multilaboratory difference between two tests (95 percent of the time) of 71 percent of the average at 12 percent loss. In other words, if an aggregate with a mean loss of 12 percent was sent to a number of reputable laboratories, one would expect them to report losses of between 7.7 and 16.3 percent (95 percent of the time). This very wide variation between laboratories has been recognized by other workers in this field. Bloem (1966) considered that the reproducibility of the magnesium sulphate soundness test was so poor as to discourage any attempt at its estimation. West et al (1970) found that it was almost impossible to obtain reproducibility between laboratories in the sodium sulphate soundness test. However, Illinois Highway Department (quoted in West et al) found that they could get repeatable results within a single laboratory.

The Ministry of Transportation has recognized the defects of many of the commonly used aggregate durability tests and specifications. Over the past ten years, a major re-evaluation of the current tests and development of new tests has been underway. Much of this work is described in papers by Hudec (1981, 1983, 1984a,b,c). This paper describes the development of and correlation with field performance of one test of the many that have been evaluated. In selecting new test methods, the following criteria were established as necessary. The tests chosen should, as far as possible, have:

- an excellent correlation with performance of aggregate in actual use, and
- a low variability both within and between laboratories.

It should be remembered that durability is not an aggregate property. Thus, it cannot be directly measured. Durability is the product of a large number of factors such as: the manner in which the materials are used, the fitness of design, the degree of exposure to moisture, and the freeze-thaw and wetting and drying cycles of the pavement or structure in which they are incorporated. Powers (1955) considered that frost susceptibility of aggregate particles was not a basic property of an aggregate. Litvan (1975) agreed with Powers; he concluded that frost resistance was not a material property. If durability is not a material property, then it follows that the durability to be expected of an aggregate cannot be measured by a single test.

FREEZING AND THAWING TESTS

The most commonly used unconfined aggregate freeze-thaw test in North America is the Iowa freeze-thaw test (Iowa, 1977). There are two variations of this test: sixteen cycles of freezing and thawing in a 0.5 percent water-alcohol solution or, alternatively, twenty-five cycles in plain water. The aggregates are vacuum saturated in either test.

In work for the Ministry of Transportation in 1978, P. Hudec developed a freeze-thaw test for evaluating shale. The objection to the existing Iowa test was the requirement for vacuum saturation, the large number of cycles, and the use of a water-alcohol medium. P. Hudec used a test as follows: A prepared, graded aggregate was placed in a glass Mason jar and saturated for 2 minutes in a 5 percent solution of sodium chloride. The sample was then rapidly drained and the lid sealed. The jar and contents were then cooled to about -20°C overnight, and allowed to thaw the following day. This was repeated for a total of five cycles. The jar and aggregate were then oven dried, sieved over the original screens, and the loss expressed as percent by mass of the original sample mass, in the same way as in the sulphate soundness test. A subsequent modification of the test for aggregates incorporated saturation for 24 hours (Hudec, 1983).

This test was conceived to reproduce the natural environment as much as possible: The use of a weak de-icing salt solution reproduced actual field conditions, the 24-hour saturation, and then testing in a drained but moist condition was not as severe as the Iowa test, and again thought to be more representative of field conditions.

Initial comparison of the freeze-thaw test with actual field performance of aggregate showed it was a useful test. However, the second requirement of any test, that it should have a low within and between-laboratory variation, in other words, be a precise test, had not been investigated.

In 1978, an extensive investigation of the "ruggedness" of the test was started. The purpose was to identify those variables which had a significant effect on the breakdown of aggregates in the test. In any test procedure, it is important to know the effect of making small, apparently insignificant modifications to the procedure. These small modifications may have a large effect on the final results obtained. Identifying these variables, and writing the test procedure so that their effect was removed, would result in a more precise test.

Effect of Solution Concentration

The first significant variable that was studied was the effect of solution concentration (Ganesh, 1979). It was known from earlier studies on concrete that sodium chloride solutions of about 2 to 4 percent by mass cause more damage during frost action than stronger solutions (Verbeck and Klieger, 1957). Twenty-five samples of various rock types and geological ages were collected from across Ontario. The samples were crushed, sieved, and frozen and thawed for five cycles in sodium chloride solutions of various concentrations. Table 1 shows the results. It can be seen that the greatest mean freeze-thaw loss was found at a concentration of 3 percent. Freezing and thawing in pure water invariability gave low losses. The magnesium sulphate soundness test and 72-hour water absorption test were also conducted. There was a significant correlation ($r = 0.73$, $n = 25$) between freeze-thaw loss and magnesium sulphate soundness loss ($MgSO_4$). The $MgSO_4$ loss was also significantly correlated with 72-hour water absorption ($r = 0.74$, $n = 25$). Gandhi and Lytton (1984) confirmed this relationship. They found a significant correlation between water absorption and sodium sulphate soundness loss ($r = 0.71$, $n = 32$). There was not a strong correlation between freeze-thaw loss and 72-hour water absorption ($r = 0.41$, $n = 25$).

Number of Cycles

A number of samples were subjected to freezing and thawing following 24-hour saturation in 3 percent NaCl solution. Testing was done on duplicate samples for five and ten cycles. It was found that ten cycle freeze-thaw losses were about 1.9 times greater than five cycle losses. It was decided to specify five cycles in future testing since this would reduce the testing time, but not change the order in which samples were rated in the test.

Temperature of Freezing

Three samples of low, medium, and high loss in the test were collected. These were frozen and thawed in the normal manner, but the temperature of the freezer was varied from -15 to -55°C. The results are shown in Figure 1. In this case, the temperature of the freezer was at the specified temperature before the samples were put in. In another study, the freezer was switched off at the completion of one freezing cycle, and the samples removed and thawed. The freezer was allowed to warm up with the

door closed, and only switched on at the beginning of the next freezing cycle. The results are shown in Figure 2. It can be seen that maximum losses were normally recorded at a temperature of -15 to -20°C. Lower temperatures and more rapid rates of cooling led to low losses. The reason is unknown, but may be due to too rapid a cooling in the -5 to -10°C range, where Litvan (1976), in his studies of sodium chloride-treated cement pastes, found the most amount of water moving in a response to freezing. It is worth noting that freeze-thaw breakdown of most rocks is probably not due to forces directly generated by ice formation. The damage is almost certainly due to pressures generated by unfrozen water moving through the porous body as a response to freezing in other parts of the body. There is some dispute whether the water moves away from the areas of ice formation due to the volume increase on formation of ice (Hydraulic Pressure Mechanism of Powers, 1945), or the water moves toward the area of ice formation due to the relatively low vapour pressure of ice compared to unfrozen bulk water in the pores (Litvan, 1972). Weak de-icer solutions such as sodium chloride, urea, alcohol, calcium chloride, and others all increase the amount of damage due to the phenomenon by which they increase the degree of saturation of the porous body which increases the rate of movement of unfrozen fluid in the system at certain temperatures (Litvan, 1976).

Temperature Variation Within the Freezer

It was found that, in the chest and upright freezers being used, there was considerable (5°C) variation in temperature within the body of the freezer. This, in turn, significantly affected the freeze-thaw losses of samples, depending on whether or not they were located in relatively warm or cool places within the freezer. It was found that this problem could be solved by placing a fan in the freezer with the samples adequately spaced and positioned so as to allow a proper flow of air.

Rate of Cooling

The optimum rate of cooling of samples was found to be about 7.5°C per hour (Figure 3).

Test Procedure

Following ruggedness testing, a final test procedure was written (MTO LS-614; see Appendix). A summary of the test procedure is given below. Oven dry samples of coarse aggregate are sieved to refusal, and samples made up as shown in Table 2.

TABLE 2: GRADINGS AND MASSES OF TEST SAMPLES

Pass	Retained	Mass, g
19.0 mm	13.2 mm	1250
13.2 mm	9.5 mm	1000
9.5 mm	4.75 mm	500

These samples are placed in autoclavable plastic Mason jars, and filled with a 3 percent by mass sodium chloride solution. After 24 hours, the samples are quickly drained, lids screwed on, and placed in a -18 ±2°C freezer for 16 ±2 hours. They are then removed from the freezer and thawed at room temperature for about 8 hours. Before placing in the freezer for the next cycle, the jars are rotated or shaken so the particles have adequate exposure to the few ml of solution in the jar. After five cycles, the samples are washed five times with tap water. This is to prevent crusting of sodium chloride following oven drying at 110 ±5°C to constant mass. The samples are sieved over the original screens and the retained material weighed. The loss is expressed as a percent of the original mass of the sample. A weighted loss is calculated based on the grading of the original sample.

A feature of the test is a requirement to test a known reference aggregate of high freeze-thaw loss (20-29 percent, mean 24.5 percent) every time the test is conducted. A 100-tonne stockpile of reference material has been established at the Ministry of Transportation laboratory in Toronto. Laboratories that obtain results with the reference aggregate that are consistently above or below the mean or outside the range have a systematic bias. It is hoped that this will ensure that laboratories obtain consistent results with this procedure.

MULTILABORATORY STUDY

Following development of the final test procedure, a simulated multilaboratory study was conducted to look at precision following the ASTM procedure (ASTM C-802). Six technicians were each given different equipment, and asked to test three replicates of three different materials with widely different freeze-thaw losses. They each followed this program twice using different kinds of freezers (walk-in, upright, chest, domestic, etc.).

A summary of the data is shown in Figures 4 and 5. These graphs show that both the standard deviation and coefficient of variation are not constant over the range of test values, but neither are they so erratic so as to be useless. The multilaboratory precision of the freeze-thaw test is also considerably better than that of the magnesium sulphate soundness test.

CORRELATION WITH FIELD PERFORMANCE

Over one hundred samples of coarse aggregate, each weighing over 100 kg, were collected from across Ontario. The aggregates were selected based on prior knowledge of their field performance in Portland cement concrete, hot laid asphalt surface and binder course, and in granular base. Field performance was rated as good (used for many years with no reported failures, popouts, or other signs of poor durability), fair (used at least once where popouts or some reduced service life had resulted, but the pavement or structure gave adequate performance for over ten years), and poor (used once and disintegration occurred, usually after one winter, which severely restricted the serviceable life of the pavement or structure; alternatively, the aggregate was of such poor quality that, had it been used, serviceable life could have reasonably been expected to have been reduced. Aggregates that were known to be unsatisfactory because of problems such as asphalt stripping, susceptibility to polishing, and alkali-aggregate reactivity were not used in the study. The criterion for classification of the field performance was based on prior knowledge of field performance based on having adequate strength and resistance to freezing and thawing.

The samples were tested in the normal aggregate tests which, in Ontario, include: magnesium sulphate soundness loss, Los Angeles abrasion and impact loss, 24-hour water absorption and bulk relative density, and petrographic examination to classify the rock types present and amounts of good, fair, poor, and deleterious components. In addition, a large number of other physical durability tests were conducted, including the freeze-thaw test.

Correlation Between Freeze-Thaw and Sulphate Soundness

Figure 6 shows a plot of magnesium sulphate soundness loss against freeze-thaw loss. There is a significant correlation ($r = 0.88$, $n = 90$) between the two tests. A similar analysis by Hudec (1983) found a less significant correlation ($r = 0.67$, $n = 96$). Work by Ganesh (1979) found a correlation coefficient of 0.73 ($n = 25$).

DISCUSSION

Figures 7, 8, and 9 show magnesium sulphate soundness loss against percent loss in the Los Angeles abrasion and impact test. Figure 7 shows performance in structural concrete, but not necessarily concrete pavement where a different environment is found. Figure 8 shows performance in asphalt surface course, but frictional properties have not been considered. Figure 9 shows performance in asphalt binder course, a much less severe environment. It can be seen that the Los Angeles abrasion loss does not predict performance in these environments when losses are less than about 50 percent. This is in agreement with the work of Bayne in 1949. He found that both the Los Angeles and Deval abrasion tests were not correlated with field performance. The sulphate soundness test is moderately successful at predicting performance. Generally, but not always, 'poor' aggregates have high sulphate soundness losses. Conversely, a number of aggregates of good and fair performance have losses as high as 35 percent, but normally less than about 18 percent in the sulphate test. Thus, using these two tests, it is not possible to consistently or reliably determine the durability of aggregate.

Figures 10, 11, and 12 show freeze-thaw loss against percent loss in the Los Angeles abrasion and impact test. These graphs show a similar picture to those for the magnesium sulphate soundness test. The Los Angeles test does not predict performance. The freeze-thaw test is perhaps a little better than the sulphate test, but is not suitable by itself in predicting performance. Remembering that durability cannot be directly measured and that any single test by itself is unlikely to be successful in predicting durability, a test other than the Los Angeles abrasion and impact test should be examined.

Figures 13, 14, and 15 show magnesium sulphate soundness loss against 24-hour water absorption. In contrast to the graphs with Los Angeles abrasion and impact results, there is a much better discrimination between good, fair, and poor field performance. Some poor aggregates with low sulphate losses have moderately high absorptions, which helps to separate performance. Despite the improvement, there is not excellent descrimination between good, fair, and poor performance. This is not unusual, considering the significant correlation between loss in this test and water absorption. There are far too many aggregates in the field where performance ranges from good to poor.

Figure 16, 17, and 18 show freeze-thaw loss against 24-hour water absorption. In these graphs compared with Figures 13, 14, and 15, the good field encompasses more aggregates, and there are less aggregates in the good to poor field. Using these two tests, there would still be rejection of some good aggregates, and many fair aggregates in the case of concrete and asphalt surface performance. However, the freeze-thaw test, when combined with water absorption, is better at predicting performance than when the magnesium sulphate soundness test is substituted for freeze-thaw.

We consider the use of such a simple treatment as shown in these graphs to be fallacious. One 'poor' aggregate plotting with otherwise 'good' aggregates distorts and gives a misleading inference about the worth of the test or tests. The errant value may, for instance, be due to a laboratory error. The next step will be to conduct multiple regression of the freeze-thaw and absorption variables to predict a frost index as proposed by Hudec (1984). Frost index would then be compared with other properties such as measures of strength (micro-Deval test, British aggregate crushing strength, aggregate abrasion value).

CONCLUSIONS

1. The maximum loss during freezing and thawing of unconfined coarse aggregate is achieved at a sodium chloride solution concentration of 3 percent by mass and at a temperature of about -18°C.

2. The freeze-thaw test has a much better multilaboratory precision than that for the magnesium sulphate soundness test. The freeze-thaw test also has a significantly better single operator precision.

3. The breakdown of aggregate in the Los Angeles abrasion and impact test, when losses are less than 50 percent, is not related to performance in either structural concrete or asphalt surface and binder course. There are many aggregates of less than 50 percent loss in this test that are totally unsuitable for use. The Los Angeles test is not useful for consistently or reliably predicting the expected durability of aggregate.

4. The freeze-thaw test is better than the sulphate soundness test at predicting field performance, but is still far from satisfactory.

5. The freeze-thaw loss in conjunction with 24-hour water absorption are the best two tests, of those considered, at predicting field performance. The prediction is not perfect, and should be augmented with other procedures such as petrographic examination and determination of field performance.

6. Further statistical work is needed to predict the likely performance of aggregate. Such work should include multiple regression analyses of such characteristics as freeze-thaw loss and water absorption in conjunction with other tests which measure complimentary properties such as strength and resistance to wear.

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TABLE 1

Freeze-Thaw Loss in Percent
After 5 Cycles

ROCK TYPE	NaCl Concentration 0%	3%	5%	8%
Sandstone	3.4	4.9	2.9	1.7
Argillite	2.5	4.7	3.0	2.9
Diabase	1.4	0.5	0.8	0.4
Sandstone	1.1	10.3	2.7	0.7
Sandy Dolostone	6.7	27.8	24.5	10.6
Shaley Dolostone	4.4	67.3	61.6	34.3
Granite	0.4	1.1	0.4	0.7
Granite	1.2	1.4	3.1	1.8
Mica Schist	4.9	4.9	5.9	5.6
Dolostone	0.4	2.3	2.4	0.6
Shaley Dolostone	3.6	68.4	78.3	88.2
Limestone	3.0	33.0	23.1	15.0
Siltstone	6.3	38.5	41.5	21.5
Mica Schist	4.1	4.4	3.6	3.1
Shaley Dolostone	0.8	48.4	63.7	40.2
Shaley Dolostone	2.3	42.3	42.0	36.9
Limestone	5.3	11.4	9.5	4.4
Limestone	1.4	38.9	28.8	16.2
Sandstone	1.0	32.7	15.8	7.1
Limestone	1.7	42.8	35.6	14.7
Dolostone	1.0	1.5	1.3	0.3
Dolostone	0.1	10.0	6.2	3.4
Sandstone	3.1	30.6	14.1	10.2
Limestone	5.1	23.8	27.3	13.9
Limestone	1.9	7.1	4.5	3.6
MEAN LOSS	2.68	22.36	20.07	13.50

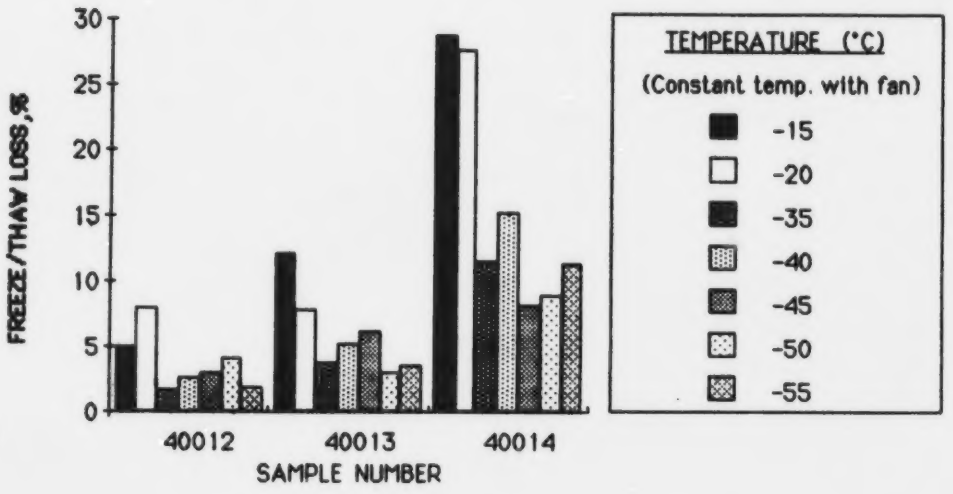

Figure 1: Freeze-thaw loss varies with freezer temperature.

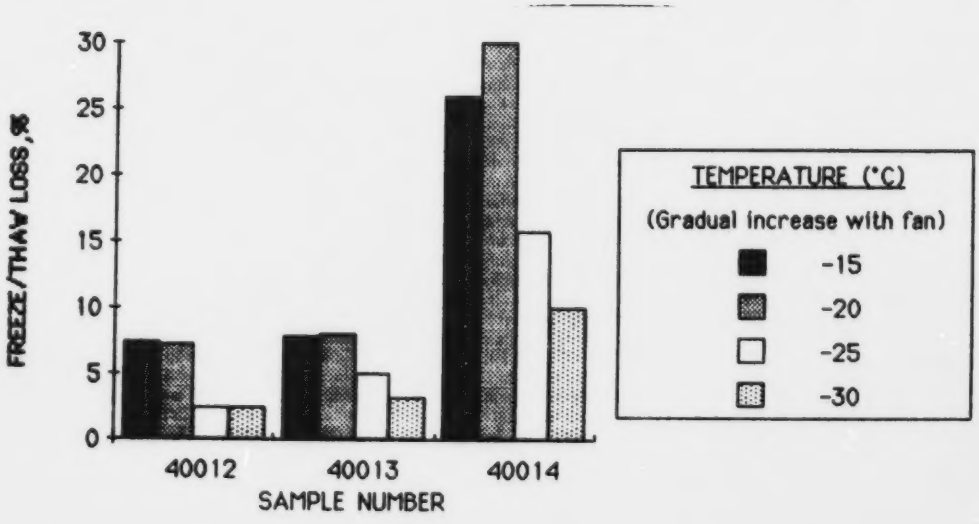

Figure 2: Freeze-thaw loss varies with freezer temperature.

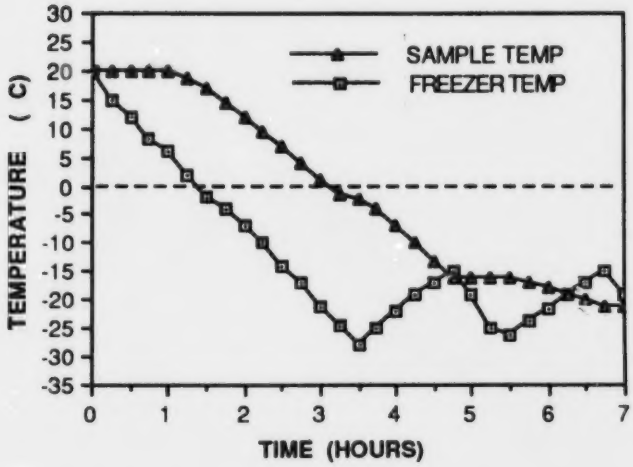

Figure 3: Drop in temperature with time for freezer air and thermocouple in center of sample container.

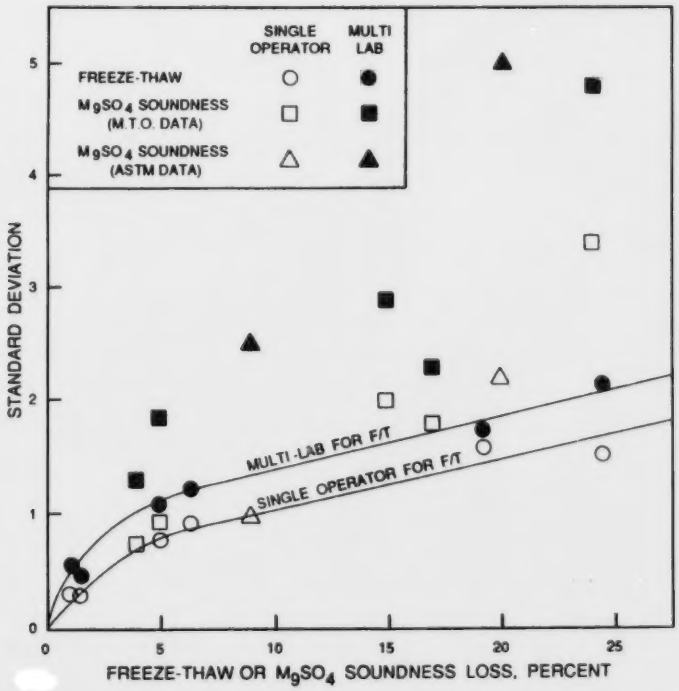

Figure 4: Standard deviation against loss in freeze-thaw or magnesium sulphate soundness tests.

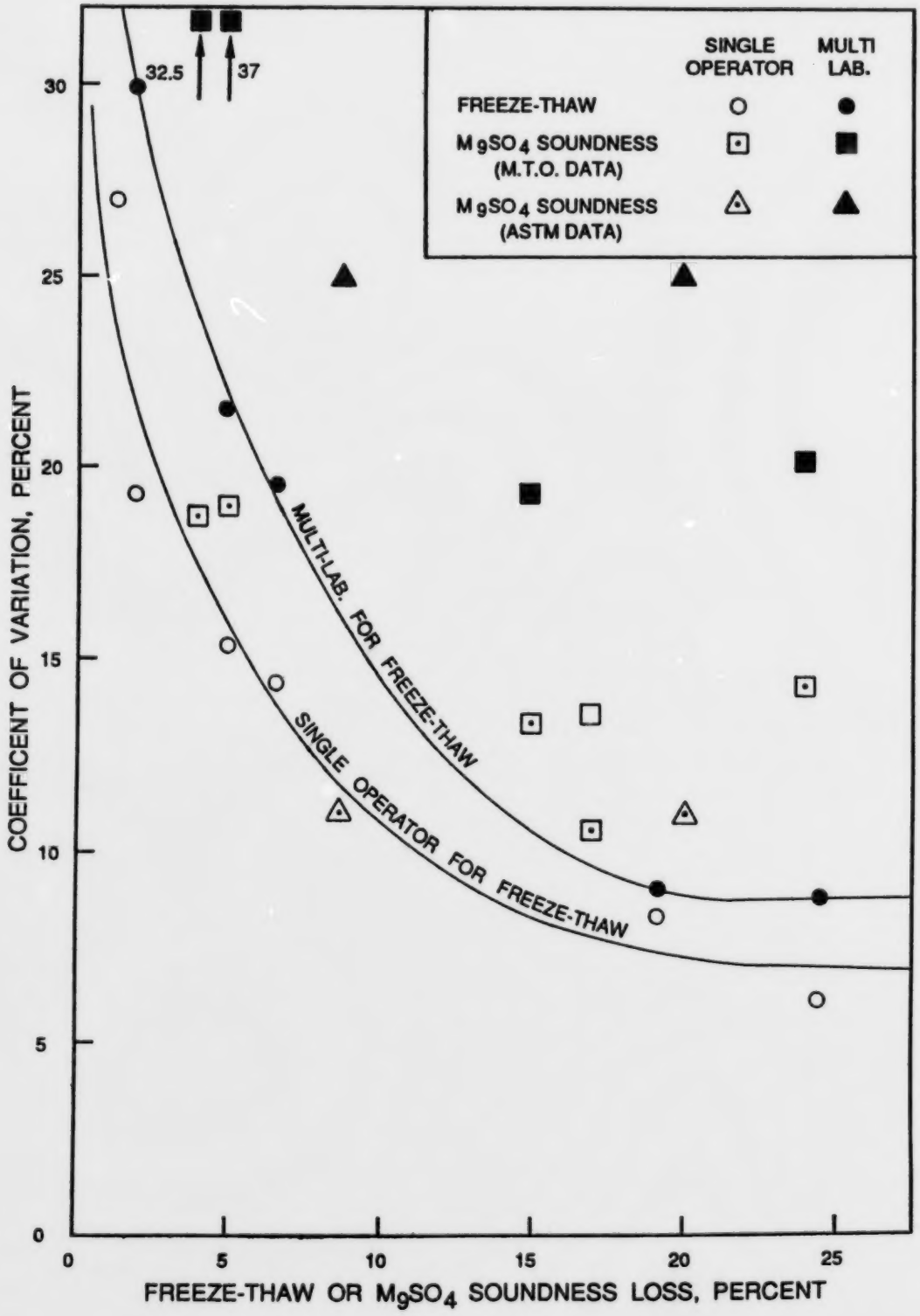

Figure 5: Coefficient of variation against loss in freeze-thaw or magnesium sulphate soundness tests.

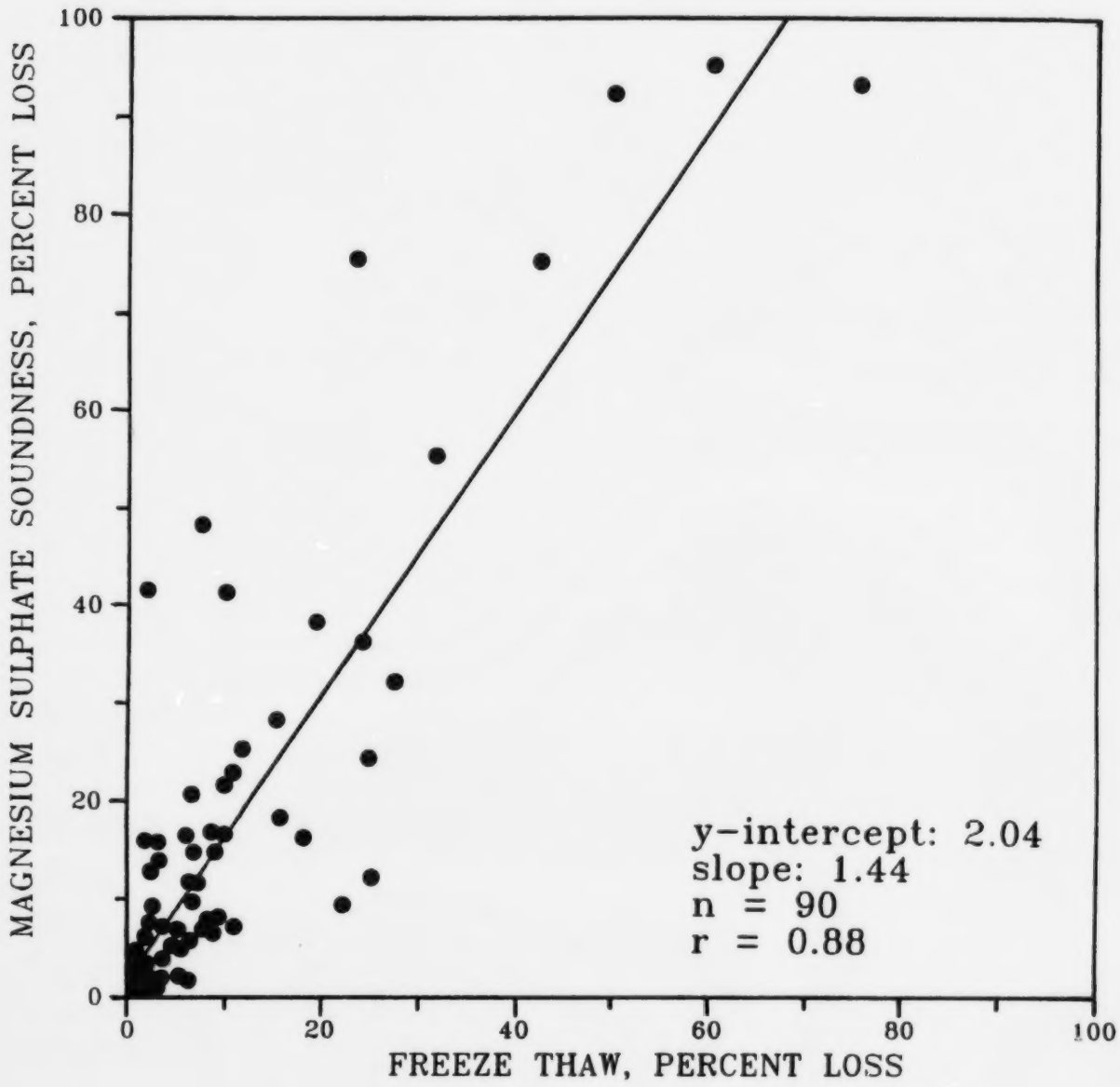

Figure 6: Magnesium sulphate soundness against freeze-thaw loss.

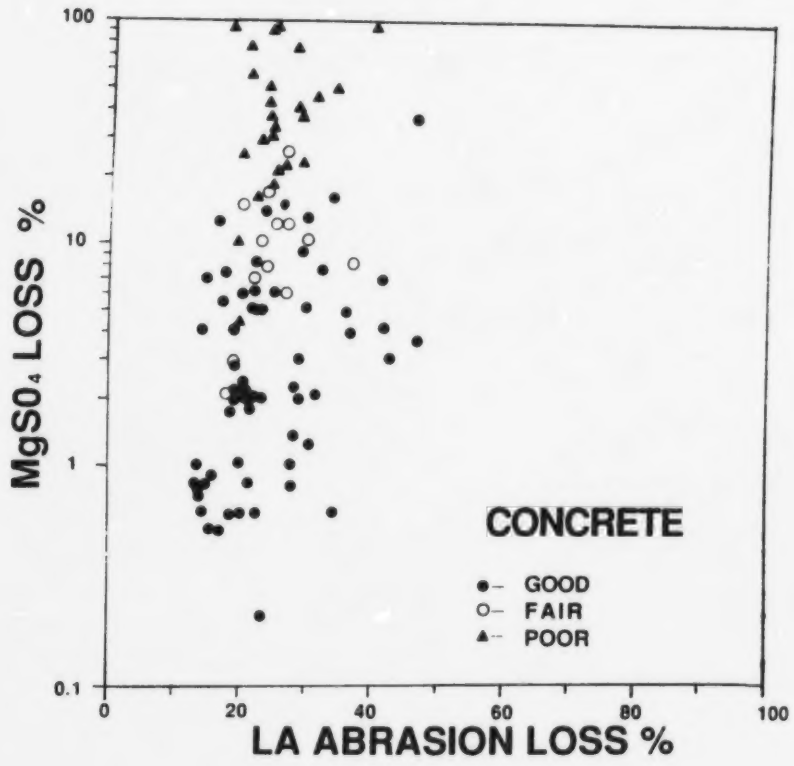

Figure 7

Magnesium sulphate soundness loss against Los Angeles abrasion and impact loss for samples of coarse aggregate of known field field performance.

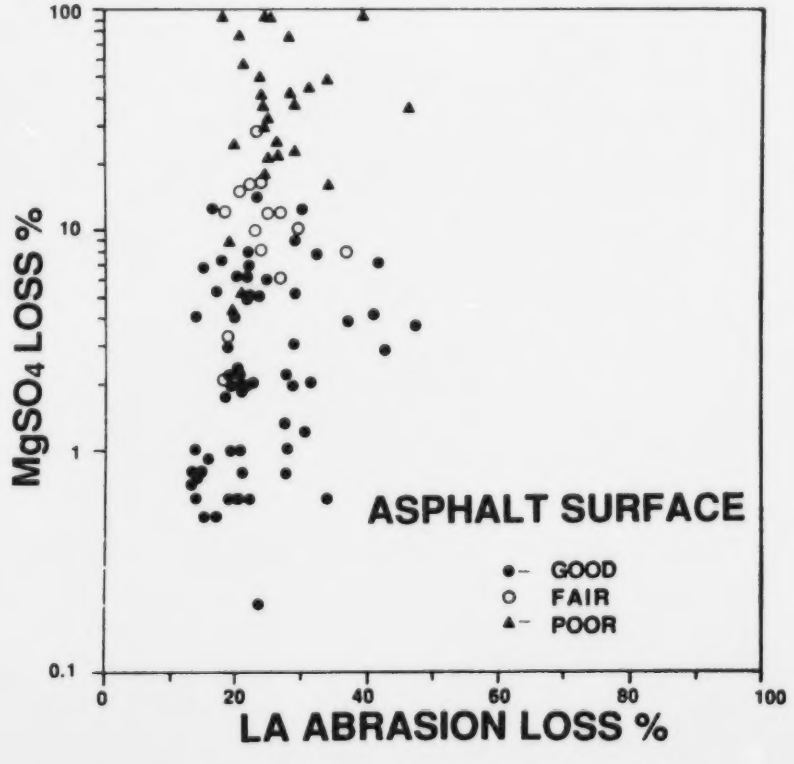

Figure 8

Magnesium sulphate soundness loss against Los Angeles abrasion and impact loss for samples of coarse aggregate of known field field performance.

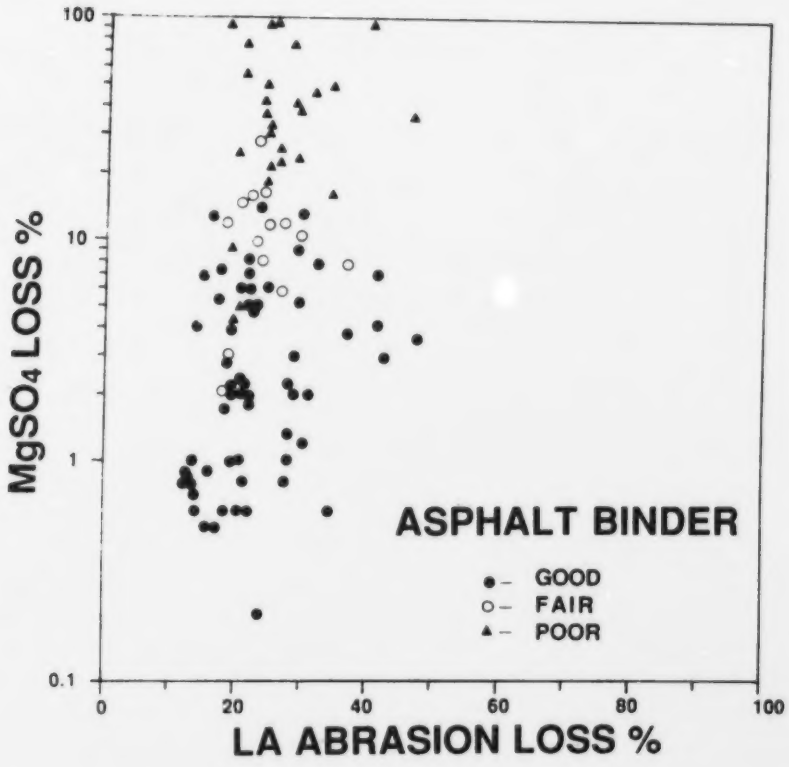

Figure 9

Magnesium sulphate soundness loss against Los Angeles abrasion and impact loss for samples of coarse aggregate of known field field performance.

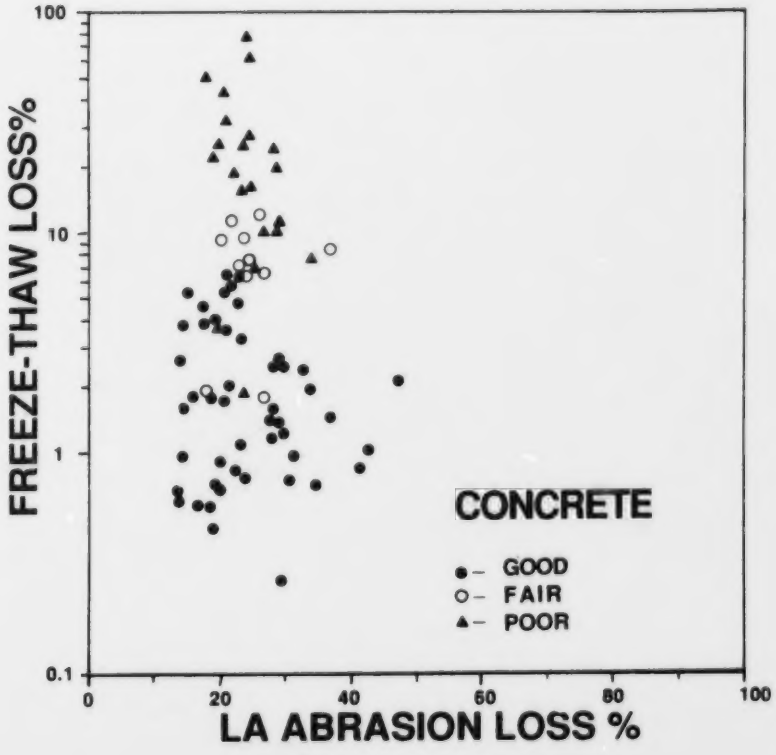

Figure 10

Freeze-thaw loss against Los Angeles abrasion and impact loss for samples of coarse aggregate of known field performance.

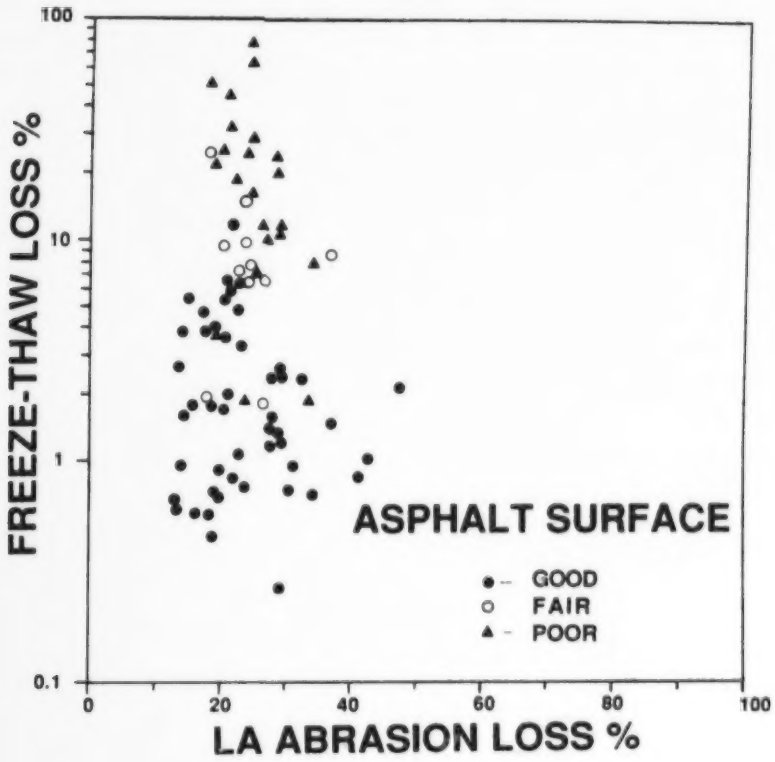

Figure 11

Freeze-thaw loss against Los Angeles abrasion and impact loss for samples of coarse aggregate of known field performance.

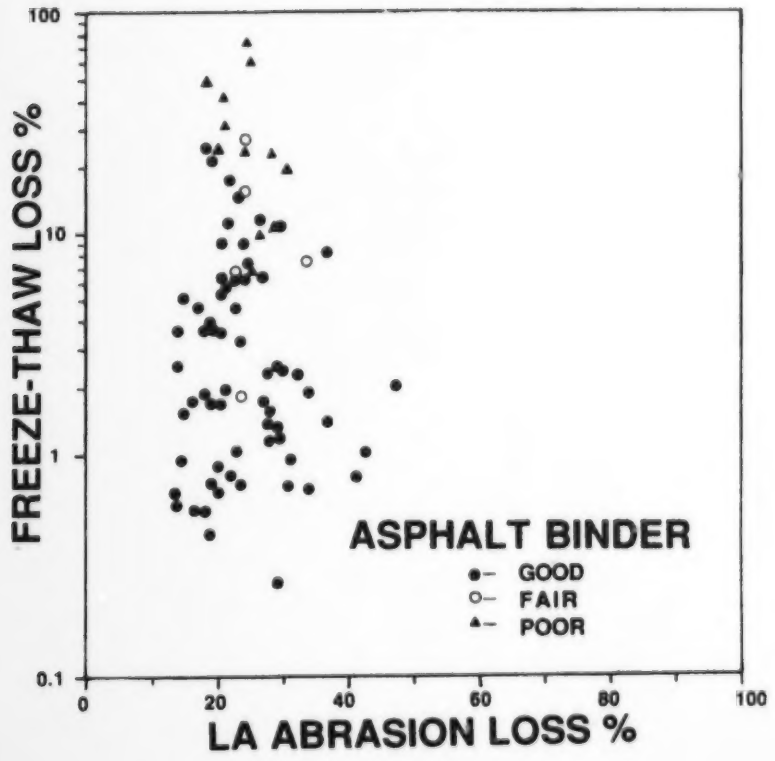

Figure 12

Freeze-thaw loss against Los Angeles abrasion and impact loss for samples of coarse aggregate of known field performance.

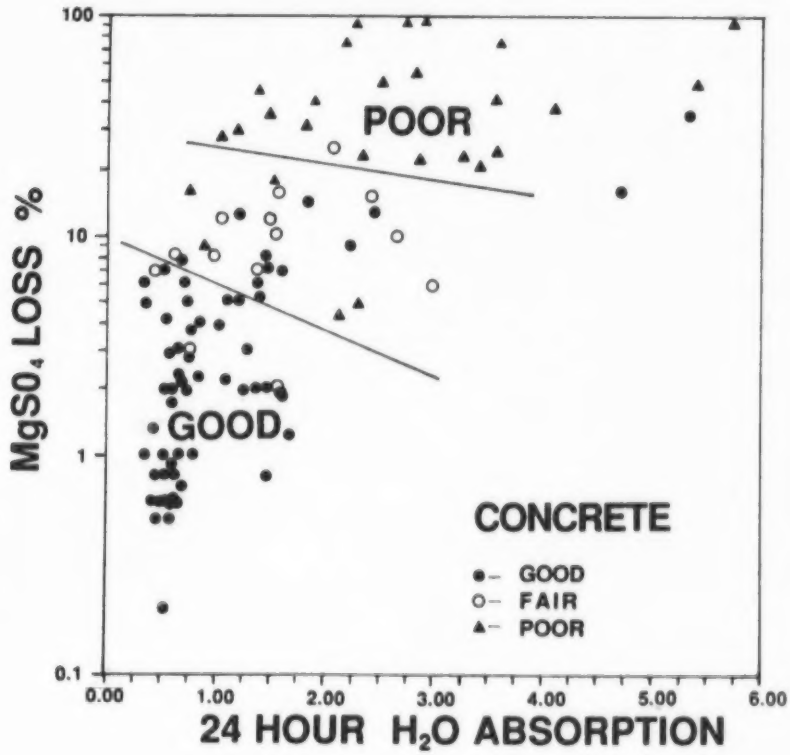

Figure 13

Magnesium sulphate soundness loss against 24 hour water absorption for samples of coarse aggregate of known field performance.

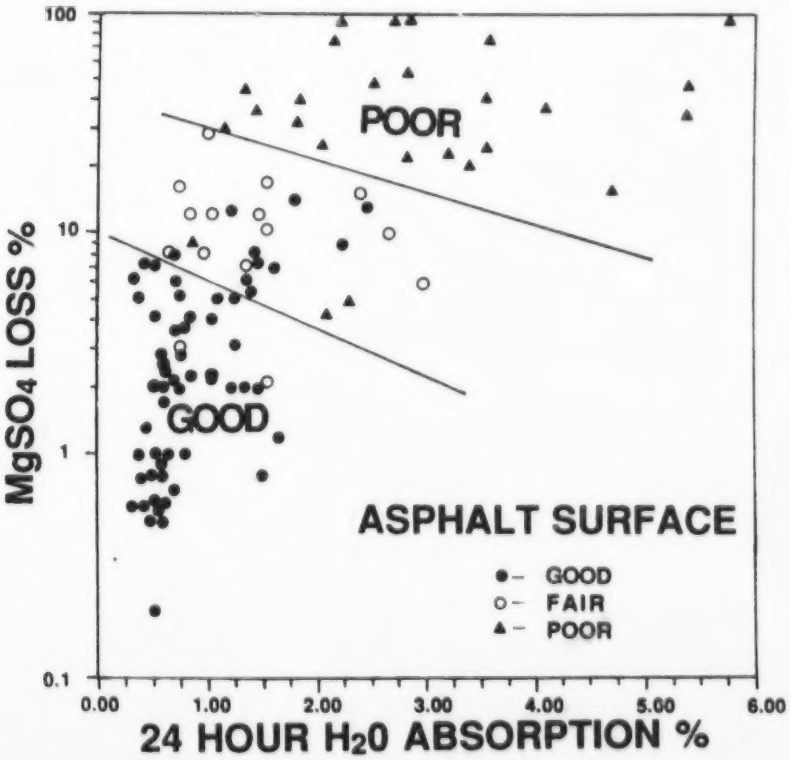

Figure 14

Magnesium sulphate soundness loss against 24 hour water absorption for samples of coarse aggregate of known field performance.

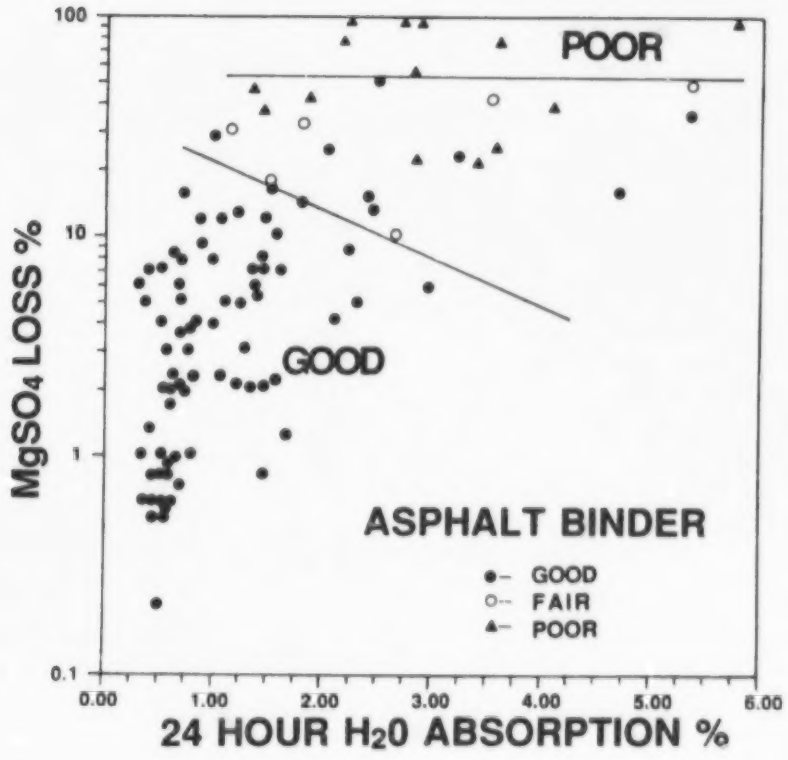

Figure 15

Magnesium sulphate soundness loss against 24 hour water absorption for samples of coarse aggregate of known field performance.

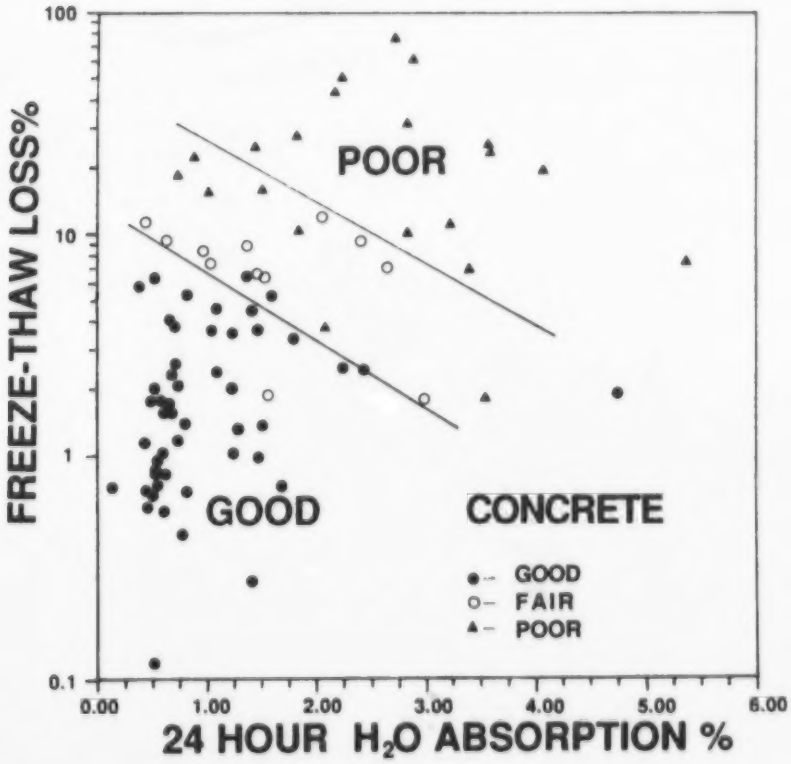

Figure 16

Freeze-thaw loss against 24 hour water absorption for samples of coarse aggregate of known field performance.

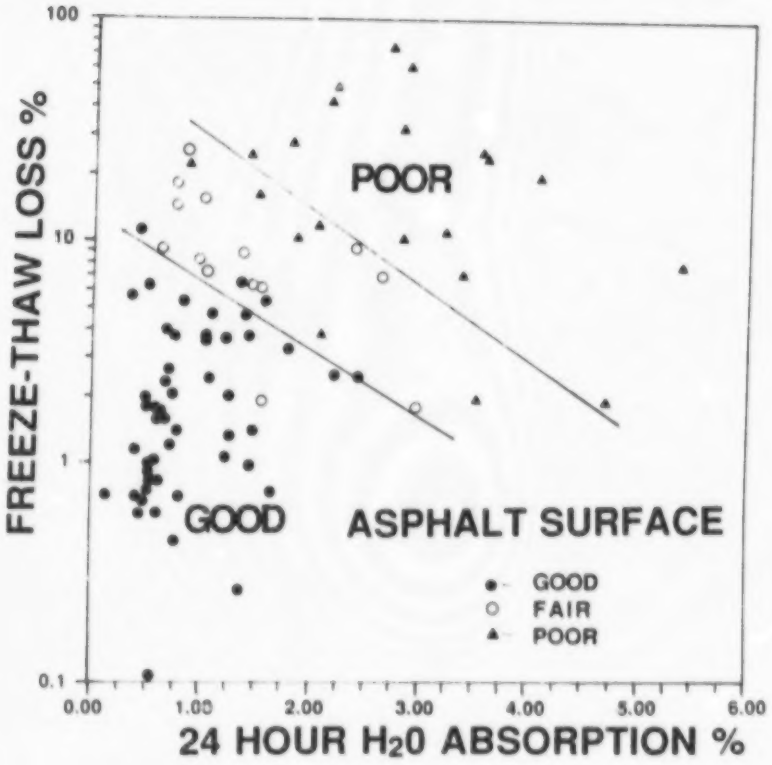

Figure 17

Freeze-thaw loss against
24 hour water absorption
for samples of coarse aggregate
of known field performance.

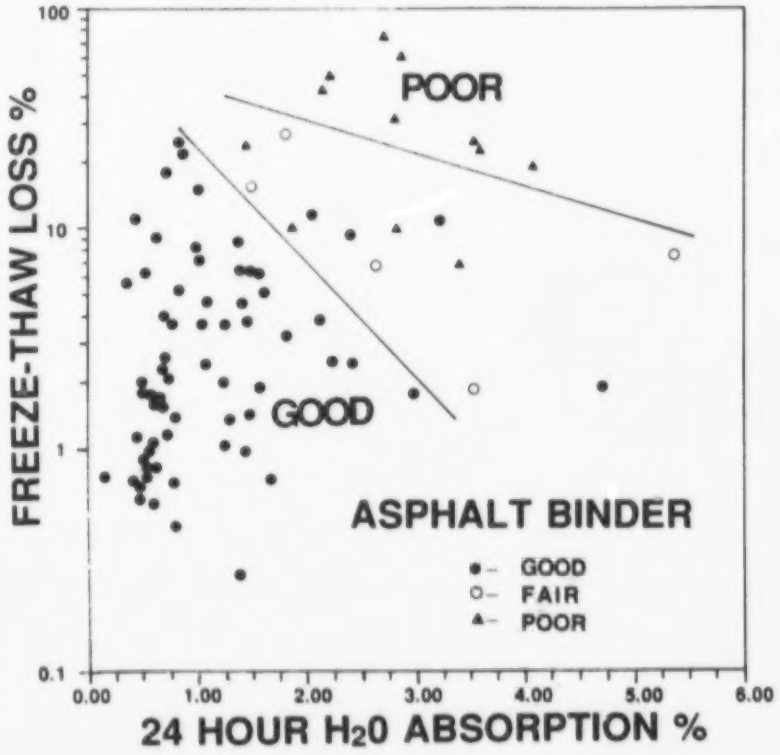

Figure 18

Freeze-thaw loss against
24 hour water absorption
for samples of coarse aggregate
of known field performance.

APPENDIX

METHOD OF TEST
FOR
FREEZING-AND-THAWING OF COARSE AGGREGATE

1. SCOPE

1.1 This method covers the testing of coarse aggregates to determine their resistance to disintegration by repeated freezing and thawing in a sodium chloride solution. It furnishes information helpful in judging the soundness of aggregates subject to freezing and thawing action, particularly when adequate information is not available from service records of the material exposed to actual weathering conditions.

2. APPARATUS

2.1 FREEZER: A freezer (chest, stand up or walk in type) capable of maintaining a temperature of -18°C ± 2°C.

The freezer must have a fan to avoid temperature differentials within the chamber.

Note: Temperature of the freezer must be continually monitored at different points within the chamber, either by thermometer or thermocouple. If thermometers are used, the bulb should be in a brass sleeve to avoid sudden temperature changes when the door or lid is opened.

2.2 SIEVES: 305 mm in diameter with square openings, and of the following sizes conforming to MTO specifications:

Sieve Sizes
19.0 mm
13.2 mm
9.5 mm
4.75 mm

2.3 THERMOMETERS: Mercury or alcohol type with a range of -35°C to + 50°C marked in 1° divisions readable to 0.5°C. All thermometers must be calibrated with an ASTM precision reference thermometer accurate to 0.1°C, at 0°C.

Note: Thermometers are available from CANLAB

Type	Model	Temperature Range
ASTM Precision	T 2352-62	-38°C to + 2°C
Lab Grade	T 2025-1C	-35°C to + 50°C

2.4 CONTAINERS: Autoclavable plastic mason jars, with air tight screw on caps able to withstand a continuous temperature of 110°C. One L jars are used for fractions retained on the 13.2 mm and 9.5 mm sieves and 500 mL jars for the fraction passing 9.5 mm retained 4.75.

Note: Autoclavable plastic mason jars are available from CANLAB

To order use: J-3030-1 500 mL
J-3030-2 1 L

Note: Containers must not come into contact with metal shelves or freezer walls because more rapid cooling will occur.

2.5 Plastic mesh baskets capable of holding four 500 mL jars or two 1 L and one 500 mL jars, and suitable wooden or plastic spacers placed between them to keep the jars from coming in contact with each other. The baskets should be stackable with sufficient clearance for the larger jars.

2.6 BALANCE: A balance or scale accurate to within 0.1% of the sample mass or 1 g, whichever is greater, over the range required for the test.

2.7 MECHANICAL CONVECTION OVEN: Capable of maintaining a temperature of 110°± 5°C.

2.8 STANDARD REFERENCE AGGREGATE: a supply of Pittsburg quarry first lift concrete stone, available from Soils and Aggregates Section, Ministry of Transportation, Downsview, Ontario

3. PREPARATION OF SOLUTION

3.1 Prepare sufficient 3% sodium chloride solution for the aggregate to be tested. For example, dissolve 30 gm of sodium chloride in 970 gm of water. Domestic table salt is acceptable. (Studies have shown that a concentration of 3% caused the most severe freeze-thaw damage). A graduated beaker or cylinder may be used for the water using 1 gm = 1 mL, but check accuracy of volume markings. Large quantities of sodium chloride solution should be prepared to minimize any error in measurement. Prior to using the solution, the container should be agitated thoroughly.

4. PREPARATION OF SAMPLE

4.1 Aggregate for the test shall consist of material retained on the 4.75 mm sieve (coarse aggregate). Separate the sample into three fractions, shown in TABLE 1, by sieving to refusal. Weigh out quantities, as indicated in TABLE 1, of the different sizes present in the sample. If any fraction constitutes less than 5% of the original sample, it shall not be tested.

TABLE 1: GRADINGS AND MASSES OF TEST SAMPLES

Pass	Retained	Mass, g
19.0 mm	13.2 mm	1250
13.2 mm	9.5 mm	1000
9.5 mm	4.75 mm	500

Prepare the material for each sieve using 305 mm diameter sieves and sieve for 10 minutes on a mechanical sieve shaker (Gilson model #SS12R or equivalent) which gives the proper sieving action. After mechanical sieving, hand sieve each fraction over a pan, striking the edge of pan on counter top and rotating 1/6 of a turn every 25 strikes for 1 minute and 150 strikes. The retained material on each sieve is then weighed and the sample placed in the appropriate size jar. Mark a number on the jar and lid and refer this number to the laboratory number and sieve size by recording on the laboratory test data cards. Lids of jars should be marked with an arrow to indicate the direction of rotation between cycles.

4.2 Every time a freeze-thaw test is conducted, a sample of the standard reference aggregate shall also be tested. The material shall be taken from a stock supply and prepared as described in 4.1.

5. PROCEDURE

5.1 Place aggregate passing 19.0 mm retained 13.2 mm in a 1 L jar. Aggregate passing 13.2 mm and retained 9.5 mm is also placed in a 1 L jar. Aggregate passing 9.5 mm retained 4.75 mm is placed in a 500 mL jar.

5.2 Saturation of samples in solution: Jars containing samples should be filled with the prepared 3% sodium chloride solution so as to completely immerse all aggregate. Seal the jars with lids to prevent evaporation and keep at room temperature for 24± 2 hours.

5.3 Draining samples after immersion: Rapidly drain off solution by inverting each jar over a screen smaller than 4.75 mm mesh for about 5 seconds (do not remove aggregate from jars). For convenience, a screen of 1.18 mm mesh can be cut to fit inside a modified mason jar lid to facilitate draining and washing. Two or three mL of solution should remain in the jar. Seal the jars to maintain a 100% relative humidity condition.

Note: Make sure the lids are screwed on properly to form an air-tight seal. When using a lid with a screen to drain off solution or when washing, make sure that the lid is on tight and/or hold a finger against the side of the lid to prevent loss of sample as lids may not fit all jars satisfactorily.

5.4 Freeze thaw cycles: Subject the samples to five cycles of freezing and thawing. Place the jars on their sides in baskets with spacers between to separate them. Place the baskets in the freezer at -18°C ± 2°C for 16 ± 2 hrs. (usually overnight) and remove them in the morning to allow approximately 8 hrs. to thaw out at room temperature. Rotate the jars one quarter turn each cycle to ensure that all aggregate particles receive adequate exposure to the solution.

5.5 Wash aggregate after thawing at the end of the fifth cycle. Fill the jar with water and invert over sink using a lid fitted with a screen as in 5.3 above. Repeat this washing five times, without removing the aggregate from jars.

5.6 Drying samples: Remove the lids from the jars and oven dry the samples to constant mass at 110°C ± 5°C, usually overnight.

6. QUANTITATIVE EXAMINATION

6.1 After drying the samples, place the aggregate on the same sieve used in the preparation of the sample and shake in the same 305 mm sieve shaker for 3 minutes, doing each size separately.

NOTE: Sieves maybe inverted before the samples are sieved (i.e. 4.75 mm sieve at the top and 13.2 mm sieve at the bottom of the nest). Any material that passes through the 4.75 mm sieve will naturally fall through the larger sieves below into the pan.

NOTE: Further sieving after 3 minutes may lead to further aggregate loss due to mechanical breakdown. Judgement should be exercised to ensure that further sieving does not induce additional breakdown.

7. CALCULATION

7.1 Calculate the percent loss and weighted average as follows:

For each fraction,

$$\text{Percent Loss} = \frac{\text{Original Mass - Mass Retained After Test}}{\text{Original Mass}} \times 100$$

Calculate the percent loss to the nearest 0.1%.

7.2 Calculate the weighted average loss as follows: From the coarse aggregate grading of the material as received, take the retained percentage for each fraction and multiply this percentage by the percent loss for that fraction. The sum of these products (3 of them for the three sizes, i.e. 13.2 mm, 9.5 mm and 4.75 mm) divided by 100 is the weighted average percent freeze-thaw loss for the sample.

Example:

FRACTION	PERCENT LOSS	PERCENT RETAINED	PRODUCT
Pass 19.0 mm/ Ret. 13.2 mm	15.0	20.0	300
Pass 13.2 mm/ Ret. 9.5 mm	18.0	30.0	540
Pass 9.5 mm/ Ret. 4.75 mm	16.0	50.0	800
		Total =	1640

WEIGHTED AVERAGE = $\frac{1640}{100}$ = 16.4%

7.2.1 For the purpose of calculating the weighted average, consider any sizes (not tested) that contain less than 5% of the "As Received" sample to have the same value as the next larger or smaller size, whichever is present.

7.3 For the purpose of calculating the weighted average of the Pittsburg aggregate use the following grading:

SIZE	PERCENT RETAINED
19-13.2 mm	35
13.2-9.5 mm	33
9.5-4.75 mm	32

8. REPORTING OF RESULTS

8.1 Report the loss on each sieve to the nearest decimal place. Report the weighted loss to the nearest decimal place.

8.2 Report the weighted loss of the Pittsburg aggregate to the nearest decimal place. This value should fall between 20 and 29 percent, 19 times out of 20. These results should be plotted on a control chart. The mean loss should be 24.5 percent after a large number of tests. Results that are consistently above or below this value show a systematic bias within the laboratory.

9. PRECISION

Criteria for judging the acceptability of freeze-thaw test results obtained by this method are given as follows:

Single-Operator Precision

19-4.75 mm Weighted Percent Loss	Standard Deviation	Acceptable Range of two results
25	1.5	4.3
19	1.6	4.5
6	0.9	2.6
5	0.8	2.3
1	0.3	0.9

Multi-Laboratory Precision

19-4.75 mm Weighted Percent Loss	Standard Deviation	Acceptable Range of two results
25	2.2	6.2
19	1.7	4.9
6	1.2	3.5
5	1.1	3.1
1	0.5	1.4

NOTE: The figures given in Column 2 are the standard deviations found to be appropriate for the material properties described in Column 1. The figures given in Column 3 are the limits that should only be exceeded one time in twenty, by the difference between the results of two properly conducted tests on samples of the same material.

Ministry of Transportation and Communications
Ontario

FREEZE THAW TEST
COARSE AGGREGATE

DATE TEST STARTED _____

LAB. NO.	JAR NO.	MASS RETAINED						PERCENT LOSS
		13.2 mm		9.5 mm		4.75 mm		
		INITIAL	FINAL	INITIAL	FINAL	INITIAL	FINAL	

REMARKS :

PH-CC-434 88-12

TEST DATA CARD

NO.	LS-614
DATE	88-04-12
PAGE	9 of 9 Rev. No. 8